



# Microstructure and Texture Evolution during Friction Stir Welding of Austenitic Stainless Steel Single Crystal

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## 論文内容要旨

Being a solid-state process, FSW (Friction Stir Welding) avoids solidification problems associated with conventional fusion welding and thereby yields sound joints, even in materials usually considered to be unweldable. The triumph of this technology resulted in its widespread usage for joining of various structural materials and gave rise to the development of derivative techniques – friction stir processing (FSP) and friction stir spot welding (FSSW). The practical success of the friction stirring techniques necessitates a more fundamental understanding of the underlying physical processes. Thus, microstructural and textural studies are presently becoming one of the key issues in the friction stirring field.

In this context, an approach involving experiments with single crystals deserves particular attention. This method may significantly simplify the microstructural and textural observations because any grain-boundary development as well as crystallographic rotations can easily be traced and interrelated with each other.

So far, however, single-crystal experiments in FSW are still rare and, more importantly, are performed mainly on aluminum alloys, i.e., on the materials with high stacking fault energy (SFE). It would be useful to expand this approach to other structural materials for which FSW is used. For instance, there is considerable interest in the application of FSW to low SFE metals (typically austenitic steels and nickel-based alloys). Superior corrosion properties of these materials are usually degraded during fusion welding, and thus FSW is considered to be an alternative welding technique.

In the present study, FSSW and FSW process were applied to an austenite stainless steel single crystal to investigate the microstructure evolution and material flow around the tool. The microstructural features and texture development were analyzed using SEM-EBSD technique. The results of each topic can be summarized as follows.

In chapter 1 “Introduction,” recent trends in FSW were reviewed and the objective of the present study was clarified.

In chapter 2 “Microstructural evolution and texture formation at different plunge depths during FSSW process”, the

structural response of single crystal austenitic stainless steel to FSSW was observed. The material flow in the probe plunging step was mainly governed by the tool probe, whereas that in the shoulder plunging step was greatly influenced by the tool shoulder. During the probe plunging step, initial single crystal was rotating toward the probe and the SZ was dominated by deformation texture where the  $\{111\}$  (shear) plane was aligned with the probe surface and the  $\langle 110 \rangle$  (shear) direction was parallel with the tool's rotational direction (i.e., tangential to the probe surface). The grain structure evolution was dominated by continuous recrystallization, which broke down the single crystal into ultra-fine grained microstructure with a mean grain size of  $\sim 0.2 \mu\text{m}$ . During the shoulder contacting step, initial single crystal deformed to be rotated toward the shoulder. The SZ was dominated by the deformation texture, in which the  $\langle 110 \rangle$  (shear) direction is again parallel to the tool's rotational direction, whereas the  $\{111\}$  (shear) plane is close to the shoulder surface. The microstructure evolution was characterized by the discontinuous recrystallization induced by increased heat input which coarsened the grain structure and led to annealing twinning. Discontinuous recrystallization was deduced to be mainly static in nature and occurred during the cooling cycle.

In chapter 3 "Effect of initial orientation on microstructure and texture during FSSW process", the crystal orientation dependence of microstructure development was examined in austenitic stainless steel single crystal during FSSW. EBSD measurements revealed that the microstructural behavior of the single crystal essentially varied in the TZ around the rotating tool (especially  $\langle 001 \rangle$ ,  $\langle 110 \rangle$  and  $\langle 111 \rangle$  shear directions were observed in detail, respectively). This effect was attributed to variable orientation of shear plane and shear direction during FSW which principally affected slip activity of the single crystal. In the  $\langle 001 \rangle$  shear direction, it appeared from the grain structure features of the TZ that relatively complicated microstructure was formed and the area of the TZ in this shear direction was much smaller than that of other shear directions. This might be related to the eight slip systems having lower values of shear factor, which led to the higher strain hardening rate. In case of the  $\langle 111 \rangle$  shear direction, the single crystal was shown to be deformed in a dissimilar way. The deformed area of TZ in the  $\langle 111 \rangle$  shear direction became much broader, compared to the  $\langle 001 \rangle$  shear direction. It is believed that this feature resulted from the higher shear factor values. The relatively high magnitude of the shear factors indicates that the slip could activate more easily. In addition, the HABs fraction formed in the TZ was found to be much lower than that in the  $\langle 001 \rangle$  shear direction. It seems that the lower HABs fraction was due to the relatively fewer slip systems, since it might be difficult for dislocations to get tangled to make HABs in a simply slip system. Even though the microstructure evolution in the  $\langle 110 \rangle$  shear direction, where single slip occurs, was expected to be quite simpler than that of other directions, the developed grain structure was complicated. The initial orientation was retained in the vicinity of the SZ, indicating that this orientation was quite stable to the  $\langle 110 \rangle$  shear direction. The distinctive feature of microstructure in the  $\langle 110 \rangle$  shear direction is the formation of shear bands which were not observed in the  $\langle 001 \rangle$  and  $\langle 111 \rangle$  directions. The complicated grain structure developed in the  $\langle 110 \rangle$  shear direction could be attributed to this formation of shear bands. In contrast to the varied structural responses in the TMAZ, the initial crystal

orientation had less effect on the microstructure evolution in the SZ. That is, the anisotropy of the structural response was most pronounced relatively far from the rotating tool but it almost completely disappeared approaching the stir zone. It appears from the structure morphology in the SZ that the LAB segments gradually transformed into HABs by accumulating of the misorientation angle and all obtained textural patterns in all three shear directions was interpreted in terms of superposition of partial  $\langle 110 \rangle$  and  $\{111\}$  simple-shear fibers.

In chapter 4 “Microstructural evolution and texture formation during FSW process”, the microstructure development and textural evolution in an austenitic stainless steel single crystal during friction stir welding (FSW) was studied. The strain-induced crystal rotations were found to be induced by simple-shear deformation. With the crystal rotations, the single-crystal structure was broken up into a fine-grained polycrystalline aggregate with grain size of 2.5–4.5  $\mu\text{m}$ . Formation of the final stir zone (SZ) microstructure was governed by a competition between continuous and discontinuous recrystallization during FSW process. The distinctive feature during FSW process was that the microstructural developments on the AS and RS were slightly different each other.

In the TMAZ of AS, the LABs formation was dominant, whose traces tend to align with the welding direction. It appears from the structure morphology that the LAB segments gradually transform into HABs by the misorientation angle increasing. Combining the results of horizontal cross-section with that of transverse cross-section, the microstructural evolution in the AS can be summarized that LABs are formed to be mainly parallel with the probe’s surface along the welding direction, and then developing into HABs. However, the grain structure development in the RS was shown to differ from that in the AS. The directionality of boundaries formed in the RS was deviated  $30^\circ$  from the shear plane (tool’s surface). Furthermore, the texture pattern in the RS revealed that the  $\langle 110 \rangle$  crystallographic direction was also tilted  $30^\circ$  against the welding direction while parallel with the tool’s surface and welding direction in the AS. It is believed that this effect resulted from the transverse movement of the tool during FSW.

In chapter 5, the results of each chapter were summarized briefly.

This thesis clarified the mechanisms for the boundary formation and texture evolution in austenitic stainless steel single crystal during FSSW and FSW process. This single crystal approach was applied for the first time on an austenitic stainless steel. The experimental results revealed how the single crystal developed into severely deformed region during the tool’s plunging step and transverse movement. Even though the present study provides illustrations on the grain structure development and material flow during the friction stirring process, there are still unclear issues concerned with the responses of crystal orientations, which affected the microstructure evolution in the TMAZ. Dissimilar deformation modes in the TMAZ were attributed to the different shear factors giving an effect on the activation of slip systems. However, the mechanisms for shear band formation in the  $\langle 110 \rangle$  shear direction is not still clear, so further study is needed to provide deeper understanding

on this distinctive microstructural feature.

# 論文審査結果の要旨

摩擦攪拌接合は低入熱の固相接合法であり、攪拌部には微細な等軸結晶粒組織と局部集合組織が形成される。微細粒組織と集合組織は接合部の諸特性に多大なる影響を及ぼすため、その形成機構の解明が望まれる。攪拌部における結晶粒界および集合組織の形成機構は単結晶を用いることにより明瞭化が期待できる。本研究では、オーステナイト系ステンレス鋼の単結晶板に対して摩擦攪拌接合を行い、母材単結晶が攪拌部の微細粒組織へ遷移する領域のミクロ組織を詳細に解析して、接合過程での微細結晶粒および集合組織の形成メカニズムを系統的に明らかにすることを目的としている。論文は全編5章で構成されている。

第1章は序論であり、本研究の背景および目的を述べている。

第2章では、摩擦攪拌接合の初期段階であるツール挿入過程での微細粒組織と集合組織の形成機構について調べている。接合ツールのプローブが挿入された段階では連続再結晶が生じるが、ショルダの接触により不連続再結晶も生じることを示している。また、接合ツールの回転に沿った単純せん断により単結晶方位が回転し、攪拌部では単純せん断集合組織が形成されることを明らかにしている。

第3章では、ツール挿入過程での微細粒組織と集合組織の形成機構に及ぼす初期結晶方位の影響について調べている。活動すべり系が多くなる結晶方位の場合には、連続再結晶と不連続再結晶により微細粒組織が形成するが、活動すべり系が少ない場合には、せん断帯の導入を伴うことを示している。

第4章では、ツール挿入後走行を伴う摩擦攪拌接合過程での微細粒組織と集合組織の形成機構について調べている。ツール走行を伴う場合の微細粒組織形成も連続再結晶と不連続再結晶により特徴付けられ、攪拌部では単純せん断変形で組織が形成されることを示している。ツール挿入過程とは異なり、ツール走行を伴う場合には、微細粒組織と集合組織の形成機構に及ぼす初期方位の影響は顕著ではないことを明らかにしている。

第5章は本研究の結果をまとめた総括である。

以上要するに本論文は、オーステナイト系ステンレス鋼単結晶を用いて、摩擦攪拌接合のツール挿入過程と走行過程での微細結晶粒組織と局部集合組織の形成機構を系統的かつ詳細に明らかにしたものであり、材料システム工学の発展に寄与するところが少なくない。

よって、本論文を博士（工学）の学位論文として合格と認める。